

# Cyclic Oxidation and Hot Corrosion of NiCrY-Coated Disk Superalloys

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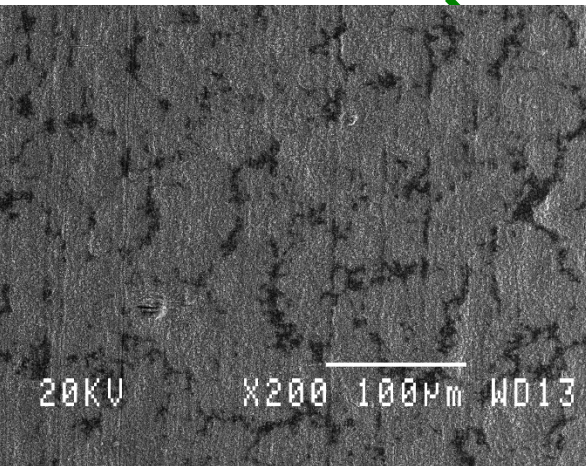
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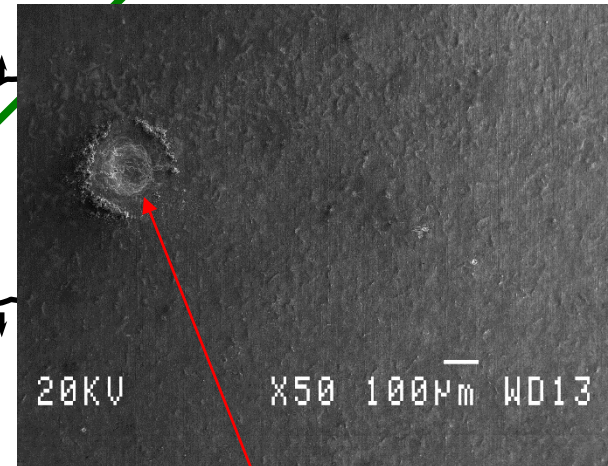
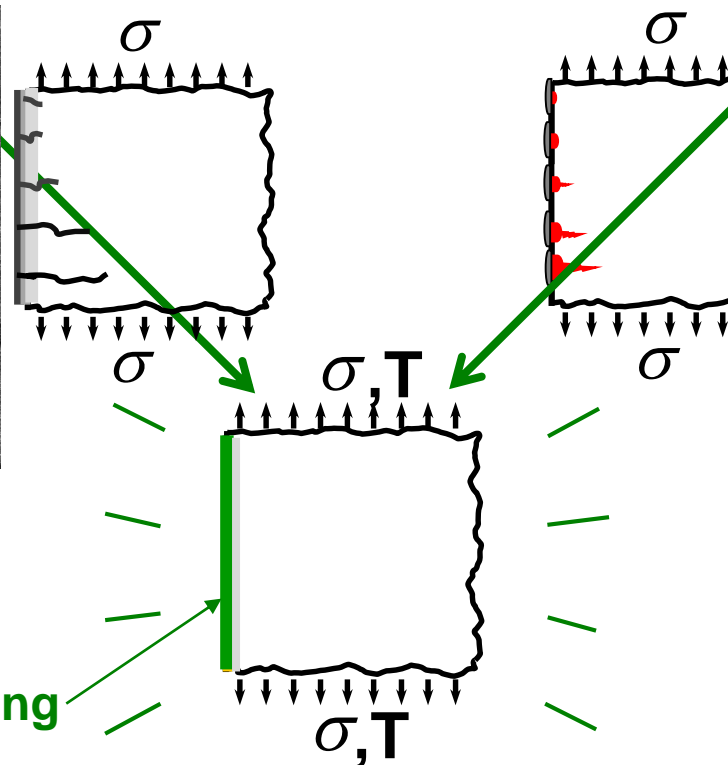
**Acknowledgements:** We are grateful to Dr. Ronghua Wei of SwRI for sputter coating, Don Humphrey for heat treating the coated pins, Jordan McCrone and Dan Gerges for assisting in the thermal cycling tests, and Rick Rogers for assisting in x-ray measurements of surface residual stresses.

# Statement of Problem

- The temperatures of superalloy disks are rising for improved efficiency.
- At temperatures around 700 °C, environmental attack by oxidation and corrosion can occur, to impair disk durability.
- Protective coatings could offer a potential solution.
- This coating would have to protect variously machined disk features.
- The coating must be resistant to mechanical and thermal fatigue→cyclic oxidation, and continue to provide corrosion protection throughout service life.



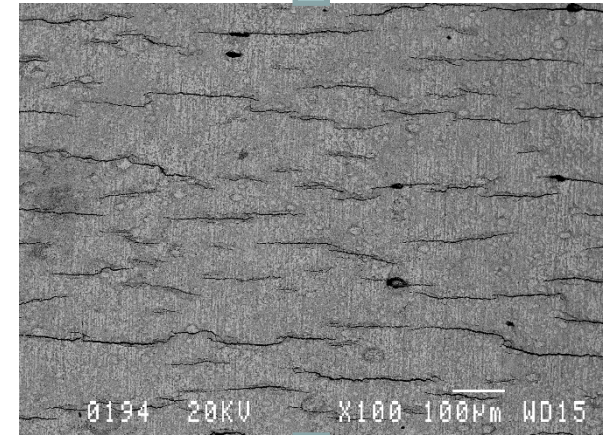
Oxide Scale on Surface



Corrosion Pit on Surface

# Early Trials

- Mechanical fatigue of at high temperatures coated specimens sometimes promoted enhanced cracking of the coating: did thermal cycling play a role in this?



## Objective

- Determine the effects of thermal cycling on oxidation and corrosion resistance of typical disk superalloys with a protective coating, for varied substrate roughness.

## Approach

- Prepare cylindrical specimens of two powder metal disk superalloys with varied surface finishes.
- Sputter coat them with a NiCrY coating.
- Thermally cycle them in air up to a high disk rim temperature.
- Remove sections for measurement of coating and cyclic oxidation layers.
- Corrode them at a high disk rim temperature using an accelerated corrosion test.

# Procedures: Materials

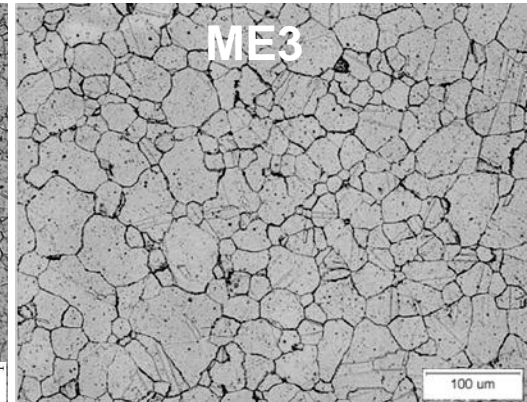
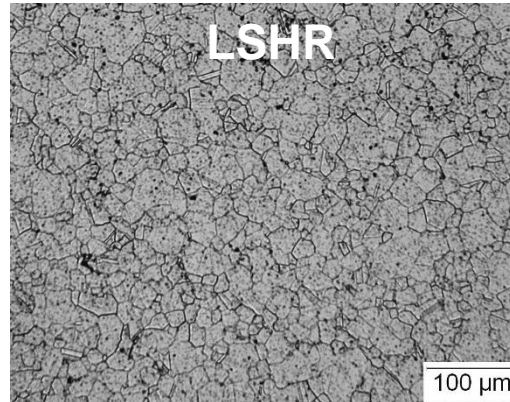
## • Powder Metal Superalloy Compositions/Microstructures

Alloy – weight percent	Al	B	C	Co	Cr	Fe	Hf	Mn	Mo	Ni	Nb	O	Re	Si	S	Ta	Ti	V	W	Y	Zr
LSHR (s)	3.54	0.027	0.045	20.4	12.3	0.1	0.0	0.0	2.71	Bal.	1.49	0.02		0.012	<.0010	1.52	3.45	0.006	4.28	<.0005	0.049
ME3 (s)	3.42	0.022	0.059	20.64	12.92				3.80	Bal.	0.91					2.30	3.58		2.01		0.051

**Supersolvus solution heat treated**

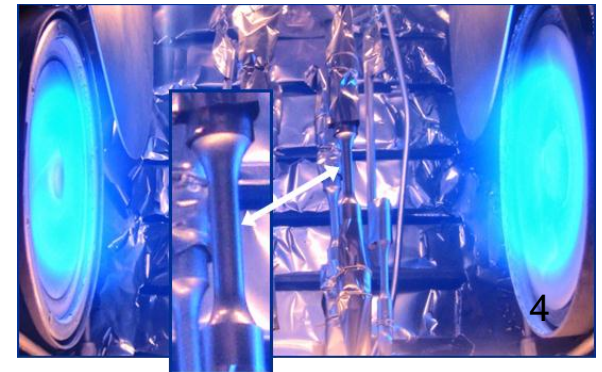
**+ 2 step aging heat treated**

- **LSHR: 15  $\mu\text{m}$  grain size**
- **ME3: 28  $\mu\text{m}$  grain size**



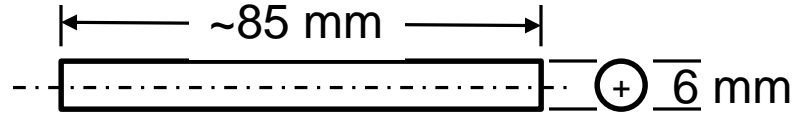
## Preparation Procedures

- **Superalloy surface machining:** Low stress grinding to cylindrical specimens, followed by abrasive polishing, vapor honing, grit blasting, or shot peening
  - Resulting average roughness ( $R_a$ ) varied from 0.24  $\mu\text{m}$  to 1.50  $\mu\text{m}$
- **Coating:** High Power Impulse Magnetron Sputtering (HiPIMS) used by Southwest Research Institute to apply the coating, using a source rod having the composition Ni-35Cr-0.1Y (wt. %); coated specimens heat treated at 760  $^{\circ}\text{C}$  for 8 h at low  $p\text{O}_2$

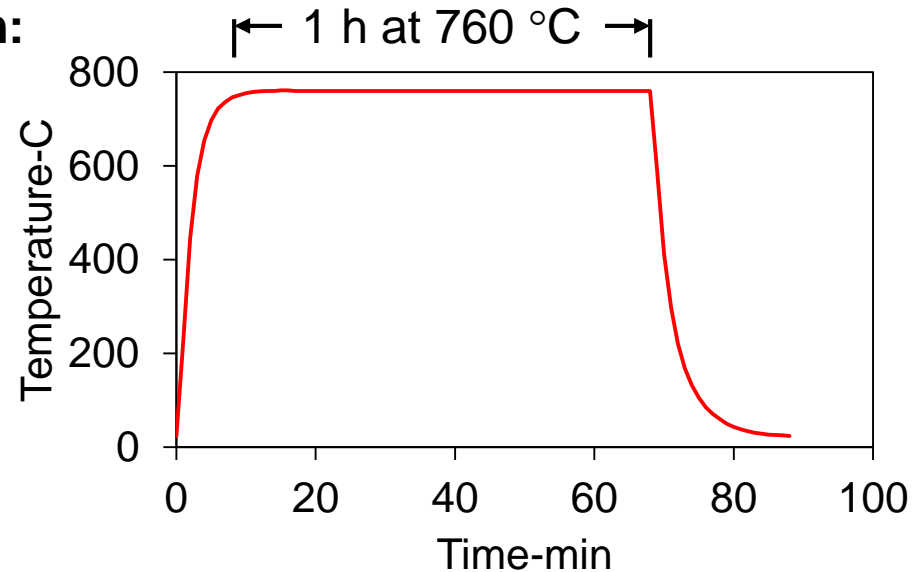
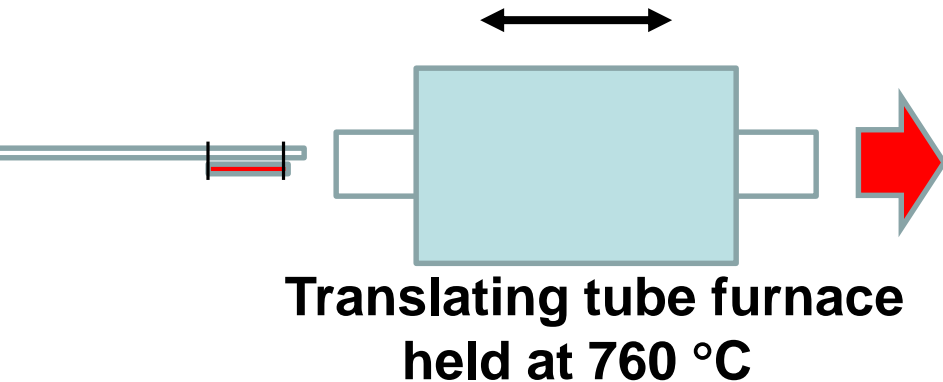


## Testing Procedures

- Cylindrical specimen



- Thermal cycling image and waveform:



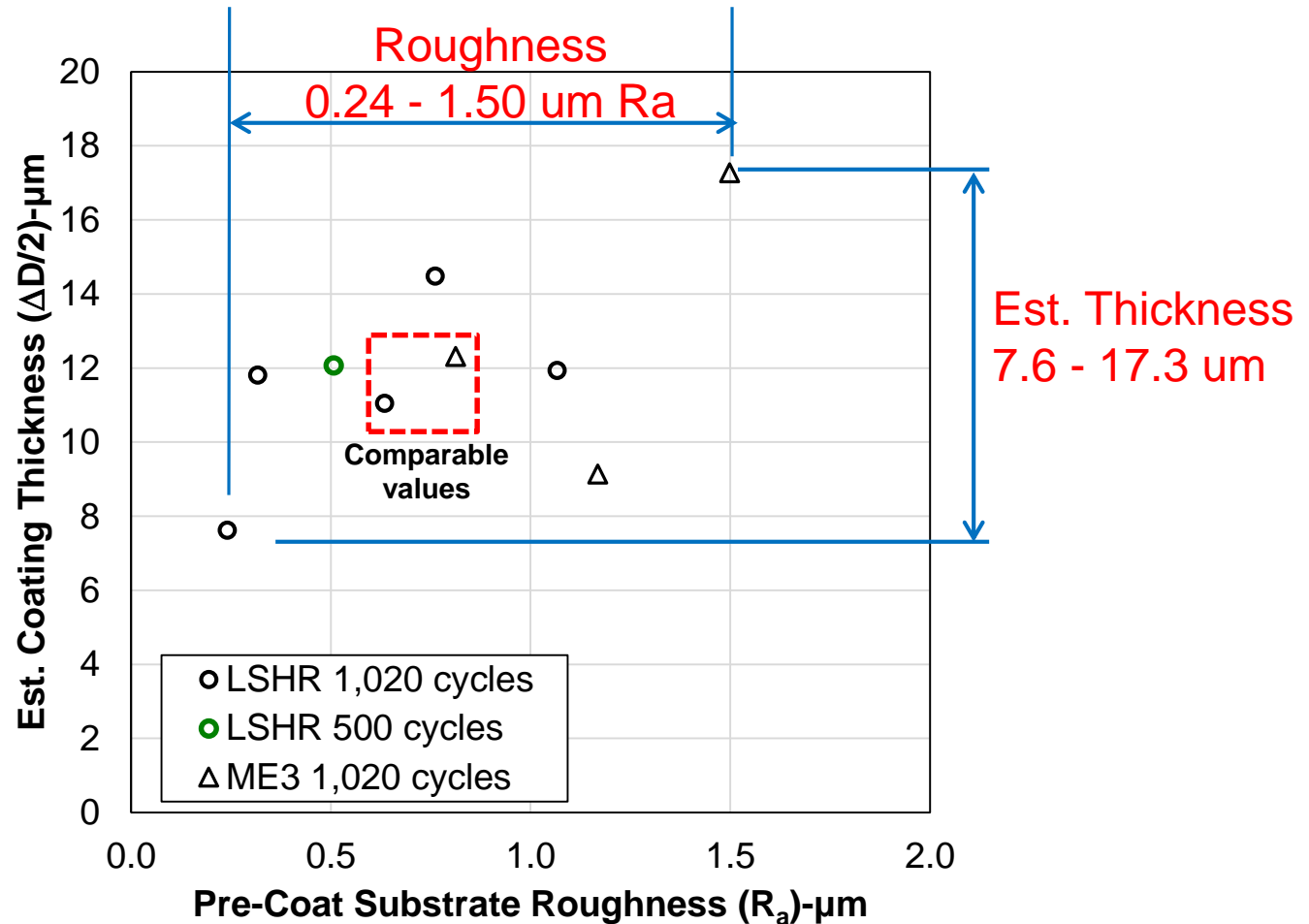
**Typical thermal cycle**

- Corrosion details:
  - Mixture of 60 wt. %  $\text{Na}_2\text{SO}_4$ -40 wt. %  $\text{MgSO}_4$  salt layer applied at 2.0 mg/cm<sup>2</sup>
  - Specimens were exposed in air at 760 °C for 50 h: this produced pits in uncoated, unexposed specimens.
- Imaging/Measurements:
  - Scanning Electron Microscopy: JEOL 6100, Hitachi 4700 Field Emission SEM
  - Coating Thickness ( $\Delta D/2$ ): Beta LaserMike Accuscan 5025-RS232 (res. 0.01  $\mu\text{m}$ )
  - Roughness: Zygo NewView 7200 (res. 0.001  $\mu\text{m}$ )



# Results: Substrate Roughness Versus Applied Coating Thickness

- A range of substrate roughness values were attained, but estimated coating thickness also varied.



- Six LSHR and three ME3 specimens were prepared and tested.
- Effects of differing LSHR and ME3 substrates could be compared near mid range (inside the dashed box above)

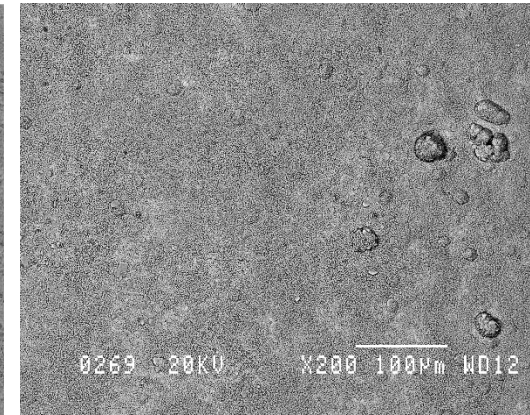
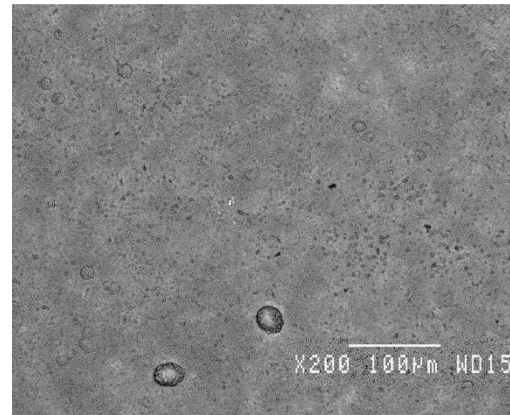
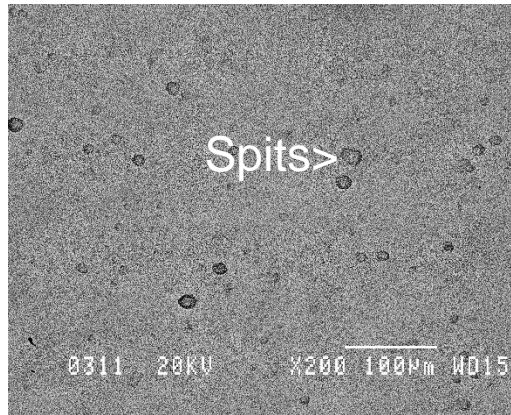
# Comparison of Coated LSHR and Coated ME3 Specimen Surfaces For Comparable Values Near Mid Range

As-heat treated

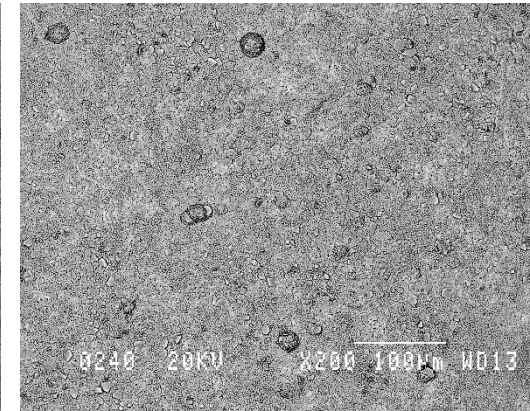
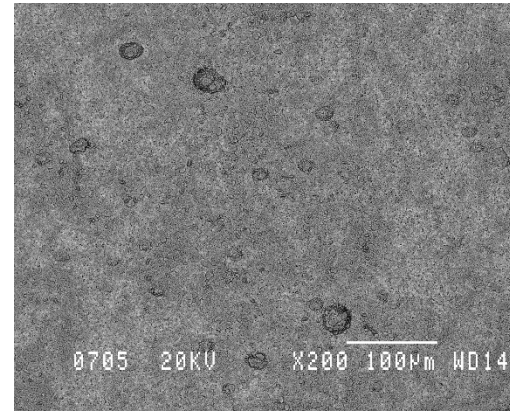
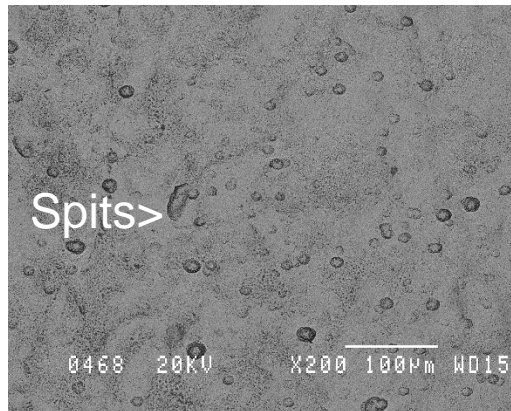
500 cycles

1,020 cycles

LSHR-3 Mid  
Roughness &  
Est. Coating  
Thickness



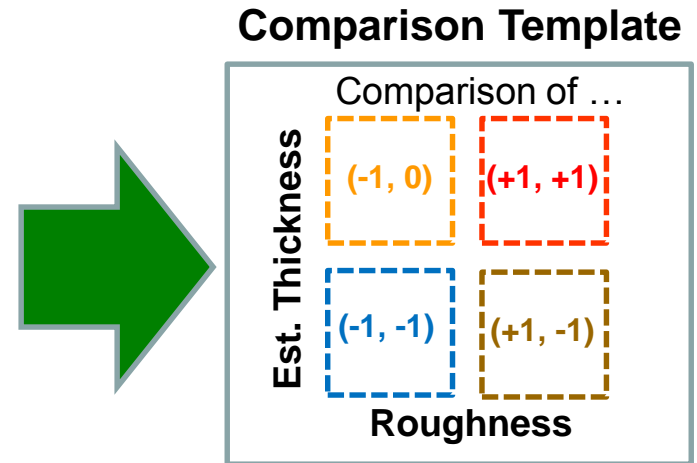
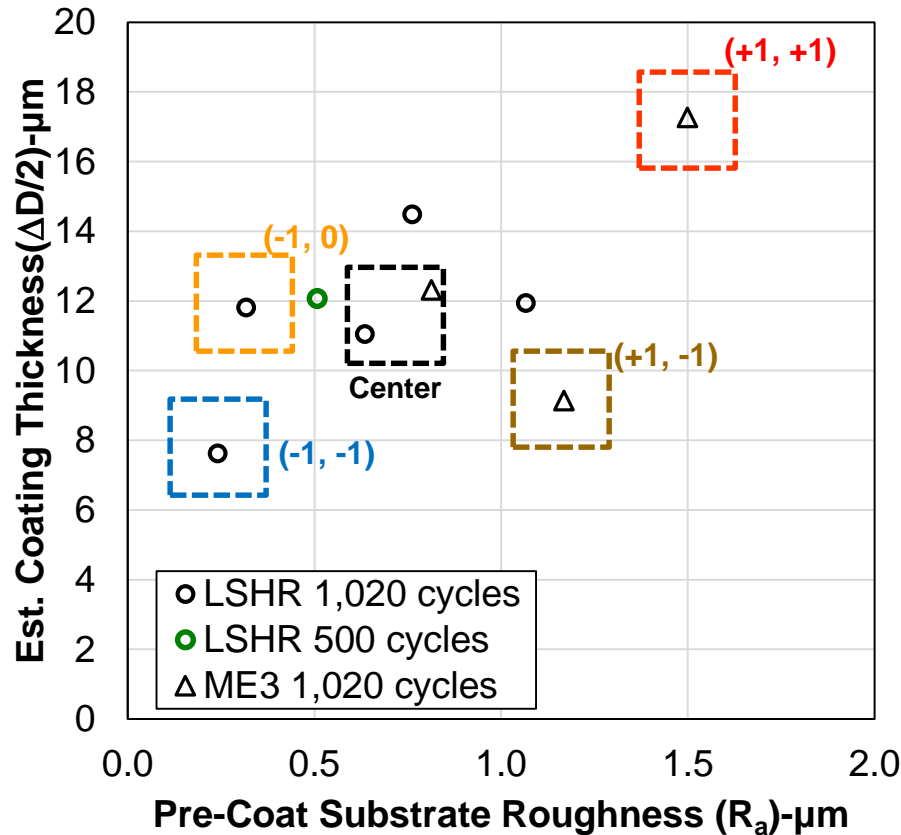
ME3-2 Mid  
Roughness &  
Est. Coating  
Thickness



- The coating compared well for LSHR and ME3 substrates at consistent roughness, estimated coating thickness, and thermal cycles.

# Substrate Roughness Versus Estimated Coating Thickness

- Together, coated LSHR and ME3 specimens gave a nice spread of substrate roughness and estimated coating thickness values

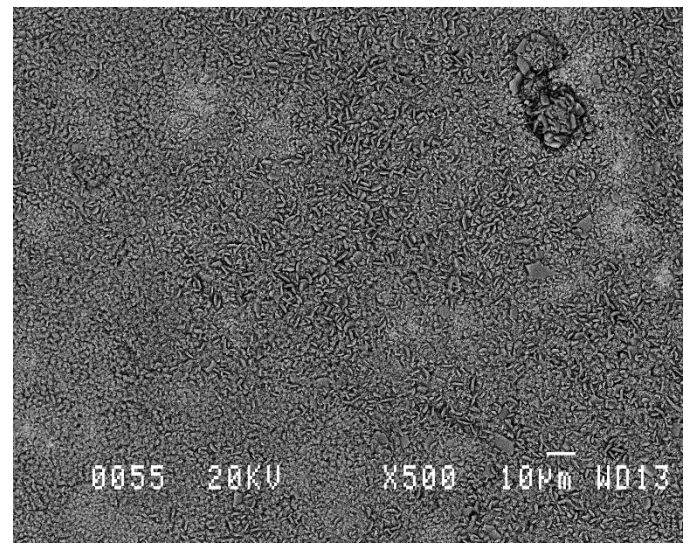
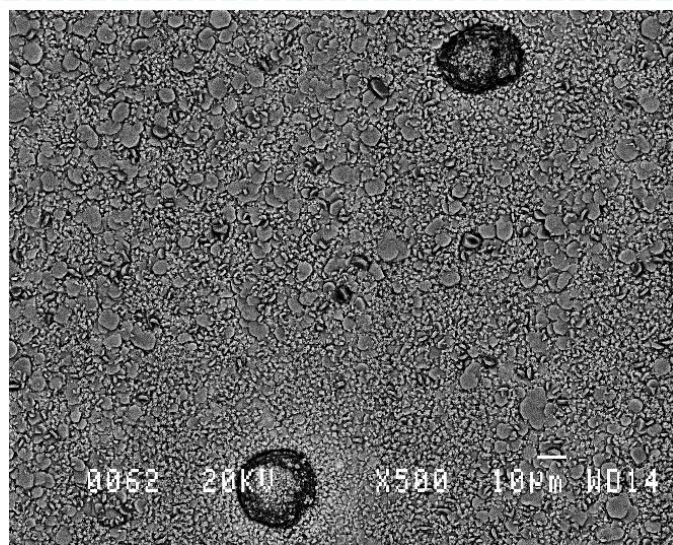
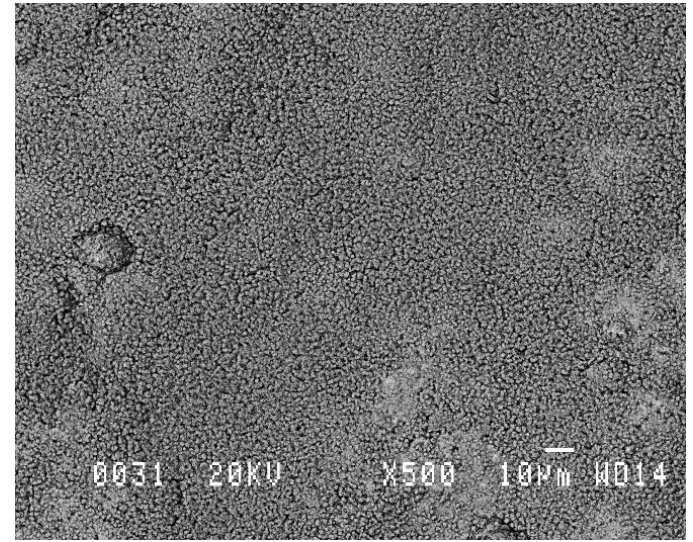
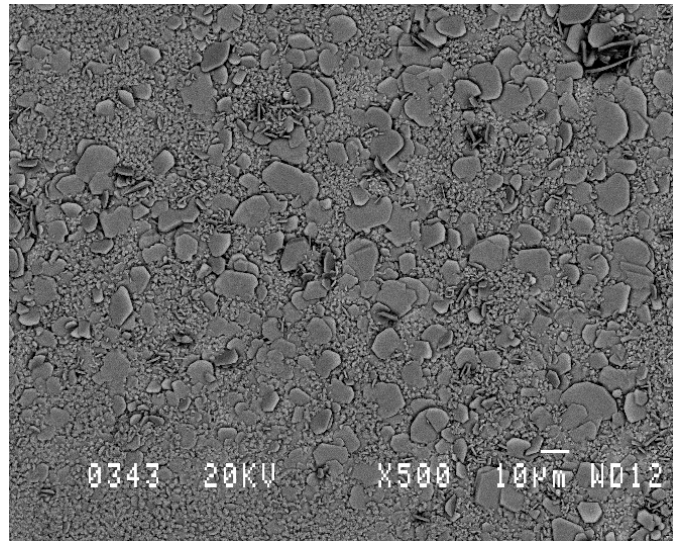


- The measured (roughness, thickness) variables for coated LSHR+ME3 were sufficiently balanced for our various comparisons.



# Comparison of Specimen Surfaces After 1,020 Thermal Cycles

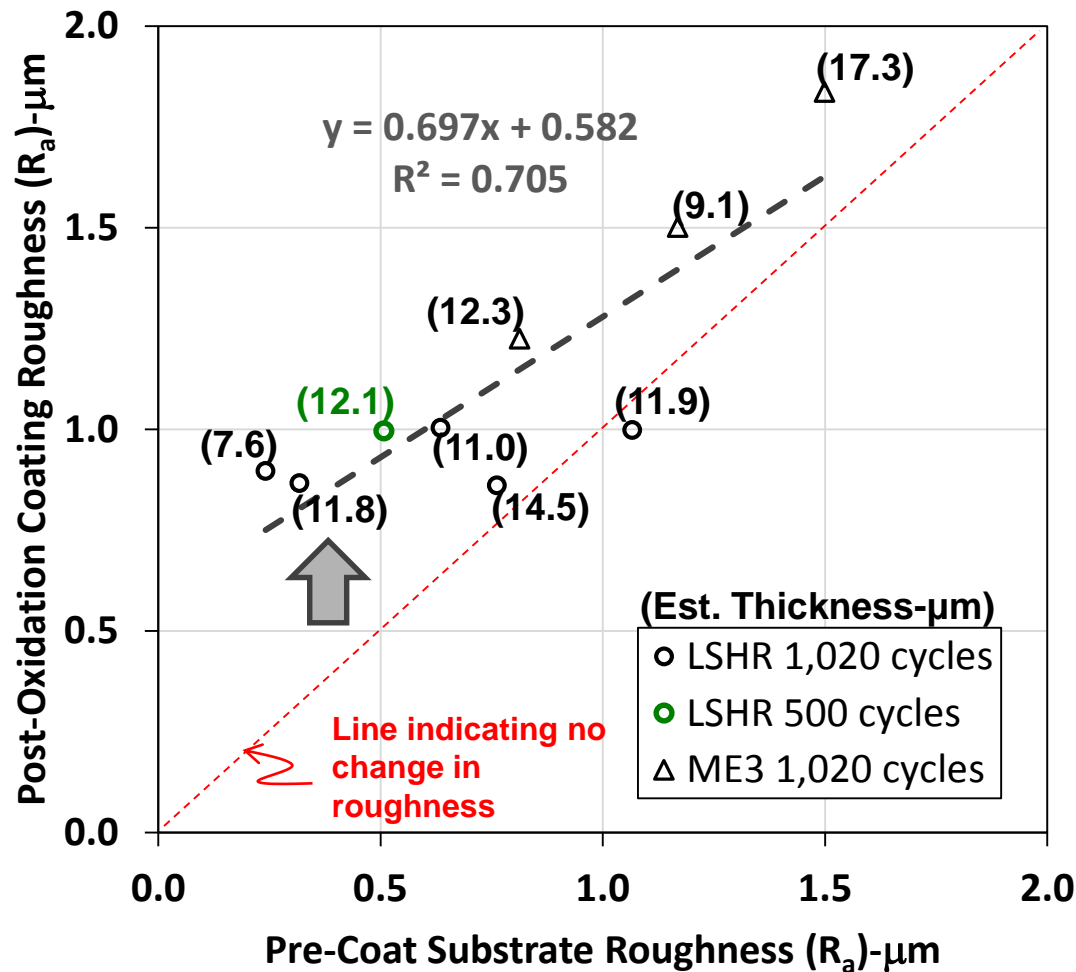
Low – Est. Coating Thickness ( $\Delta D/2$ ) - High



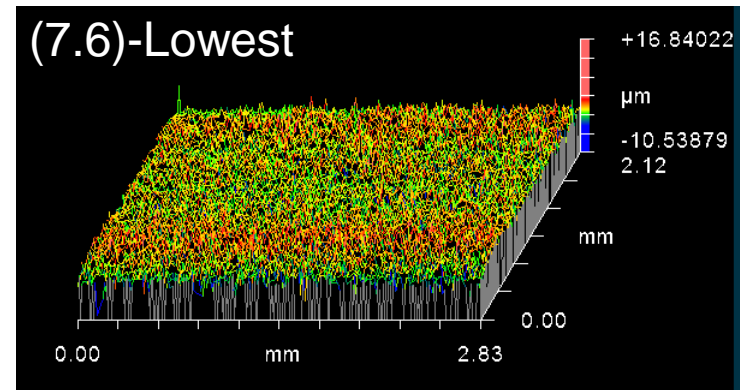
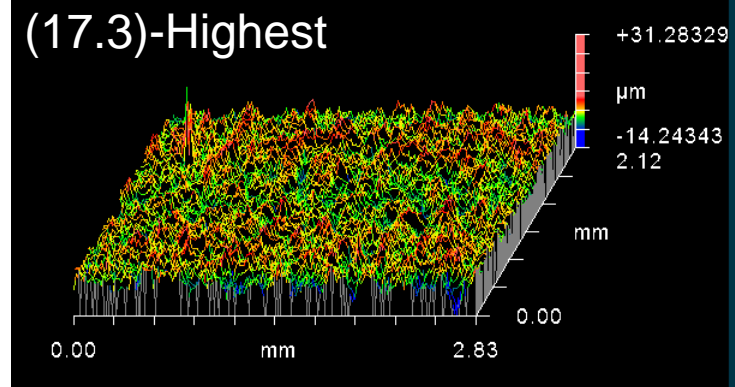
Low - Substrate Roughness - High

- No linear cracking or peeling of the coating was observed.
- No consistent effects of varied roughness and coating thickness observed.

# Pre-coat Substrate Roughness Versus Post-Oxidation Coating Roughness



## Post-Oxidation Coating Roughness

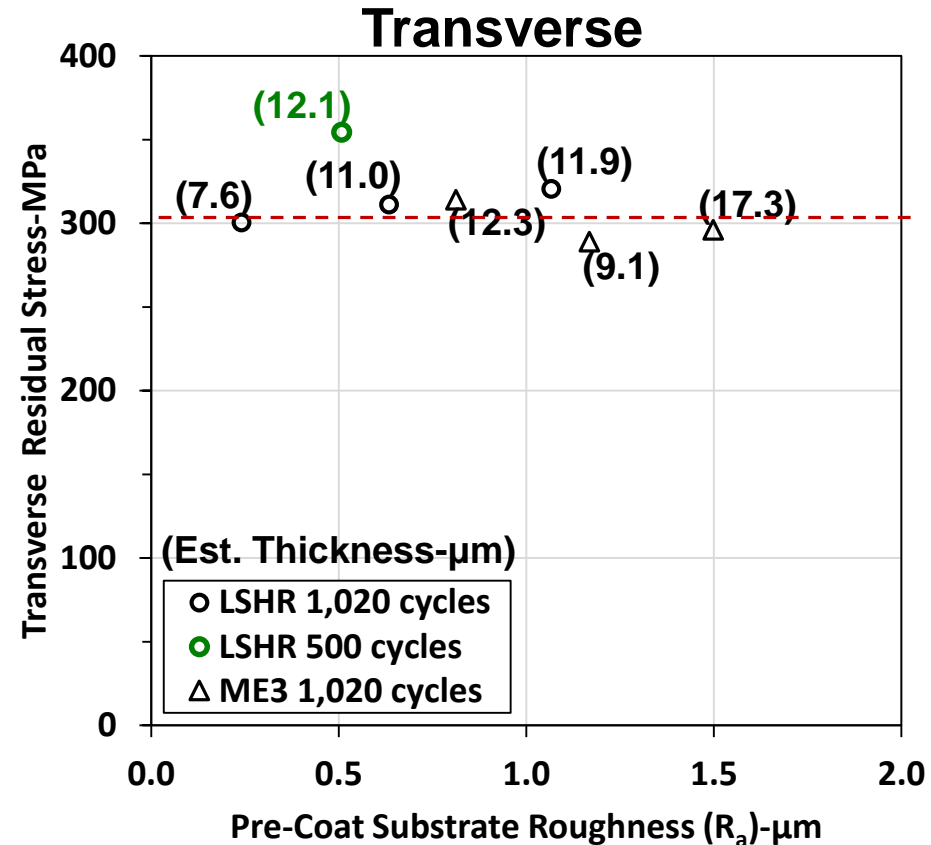
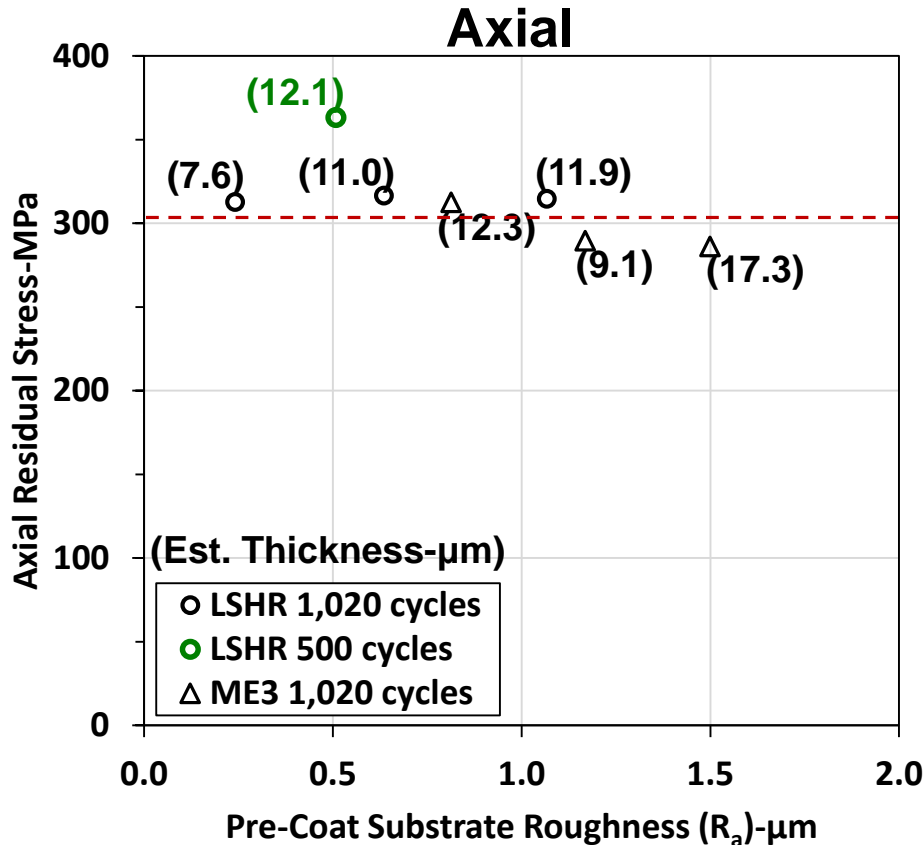


- The oxidized coating had increased roughness, yet pre-coat substrate roughness and post-oxidation coating roughness were still correlated.
- No consistent effect of estimated coating thickness was evident.



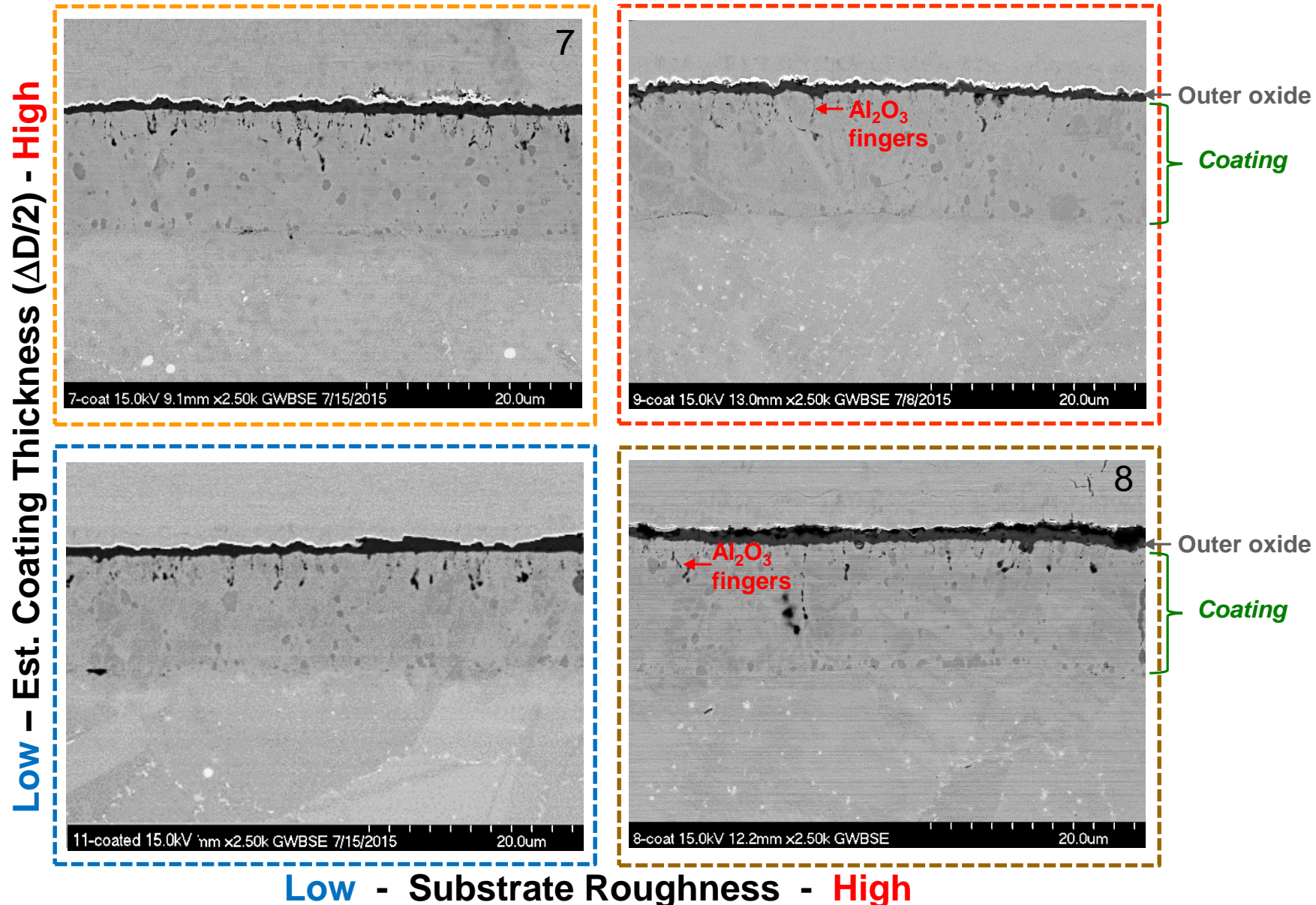
## Average Axial and Transverse Residual Stresses in the Coating

- Bruker D8 Discover X-ray diffractometer with area detector, using Mn tube on specimen target area of 1.2 mm<sup>2</sup>, (311) reflection



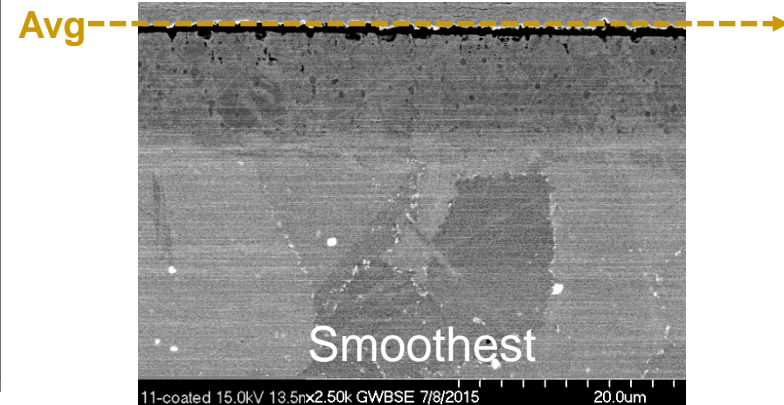
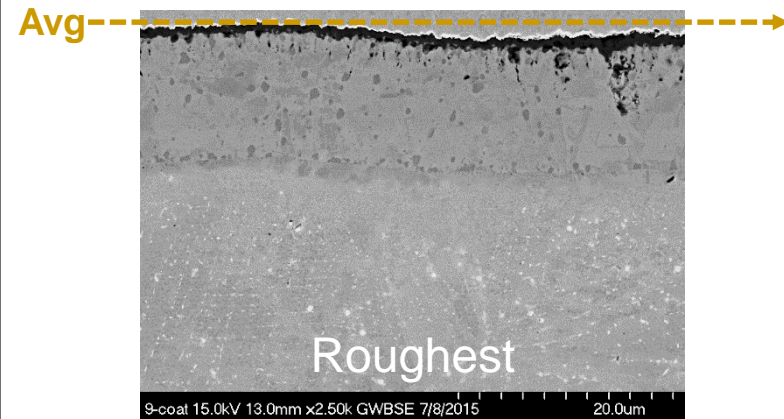
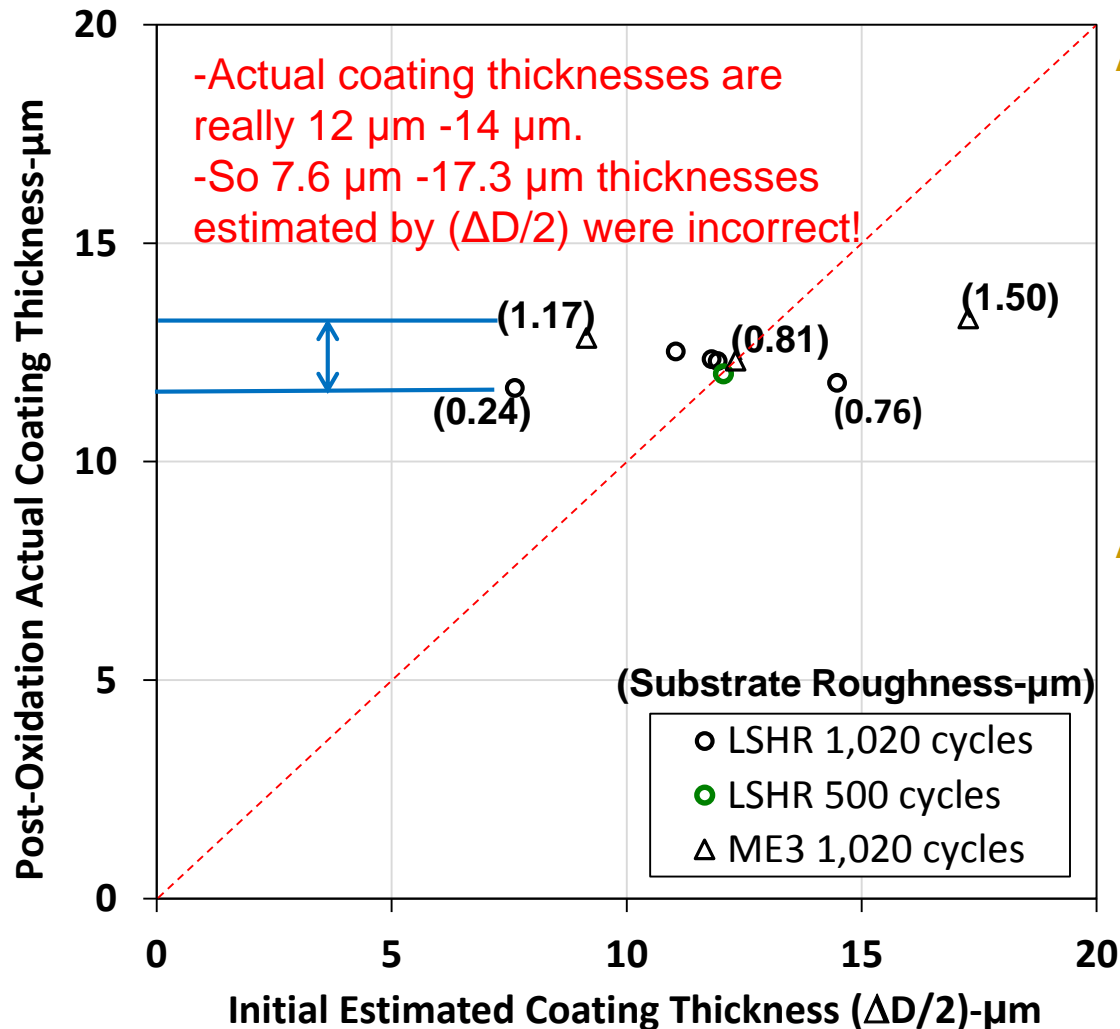
- Comparable tensile residual stresses were measured in both axial and transverse directions after thermal cycling.
- No strong effects of varied substrate roughness and estimated coating thickness were observed.

# Comparison of Coating- Oxide Layers in Sections After 1,020 Cycles



- Protective coating layer was still continuous, and mostly intact.
- Comparable remaining coating thickness was evident for these extremes.

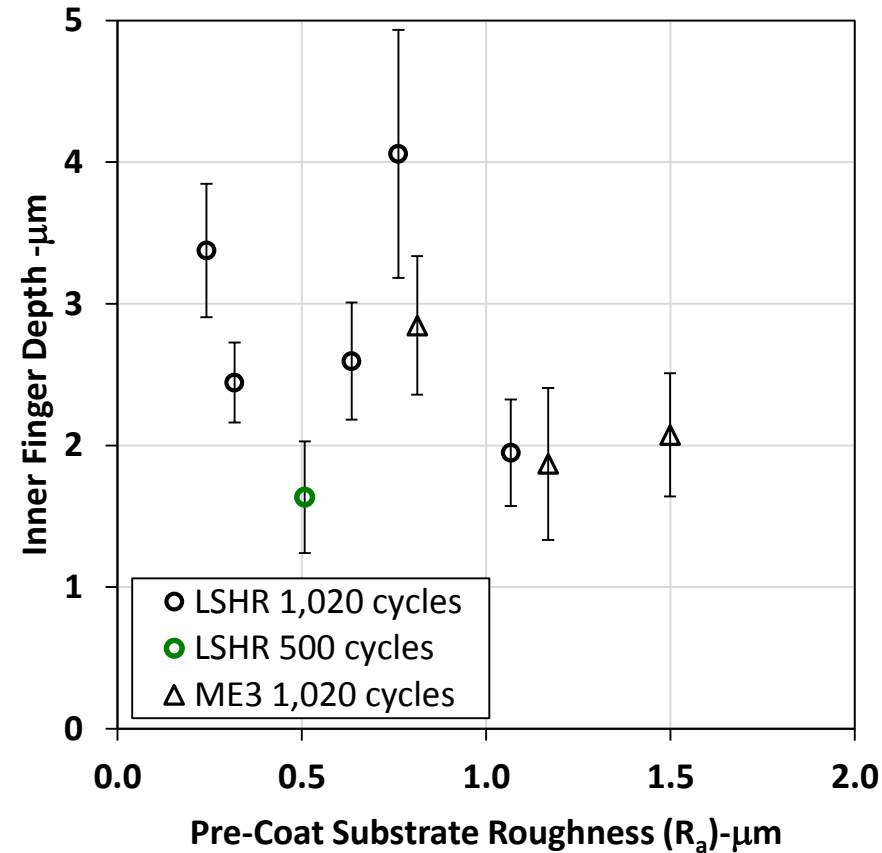
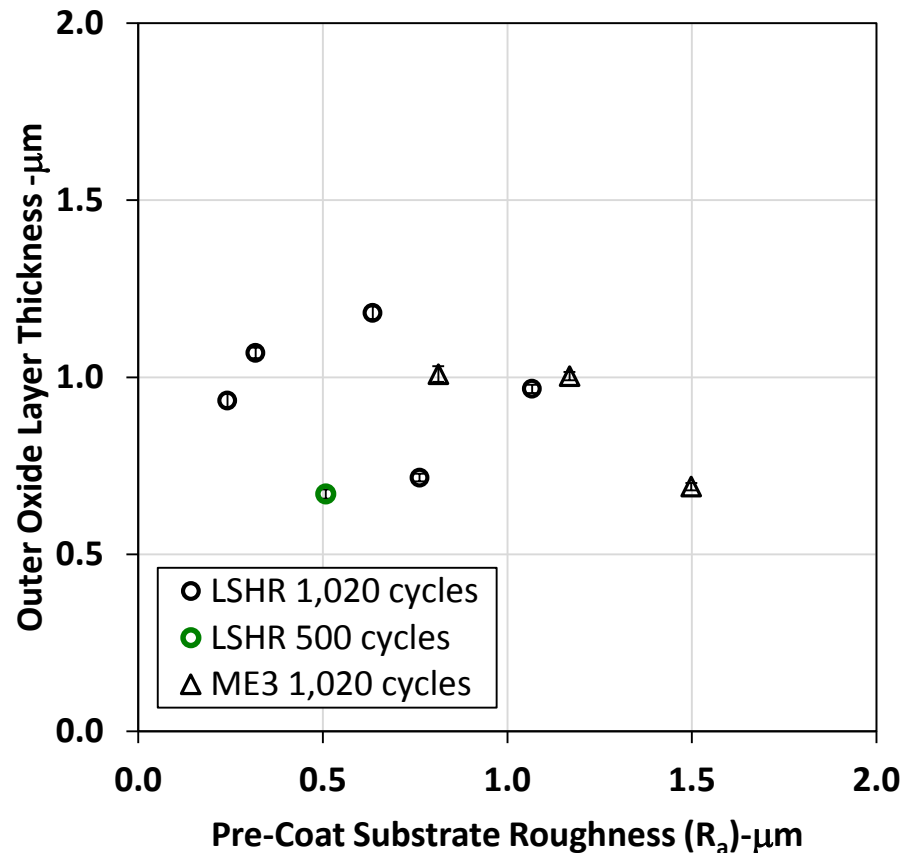
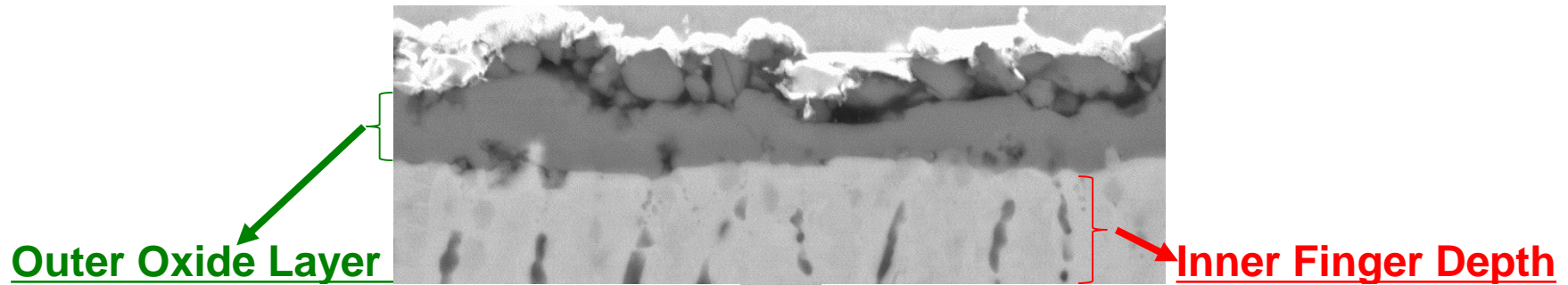
# Coating Thickness: Initial Estimated Average Versus Cycled Actual



- Estimations of coating thickness by change in dimensions ( $\Delta D/2$ ) did not only reflect coating thickness, but also other factors (e.g. roughness).
- No strong effect of varied substrate roughness on cycled actual coating thickness was consistently observed.



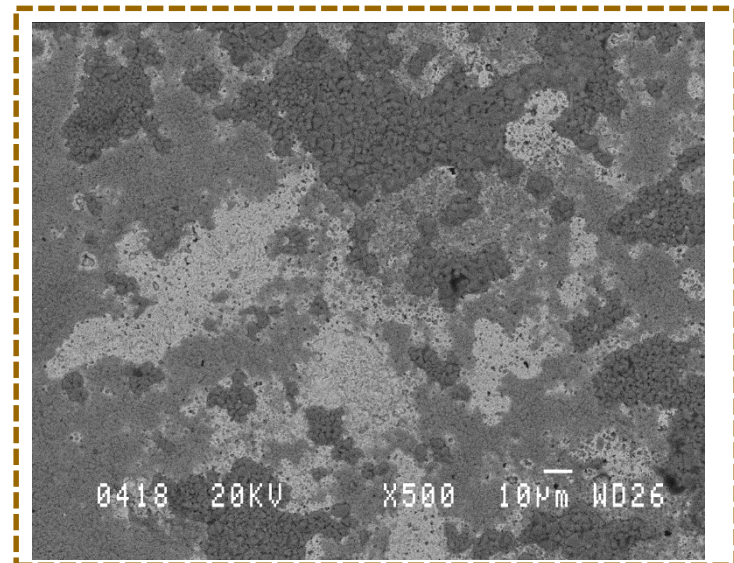
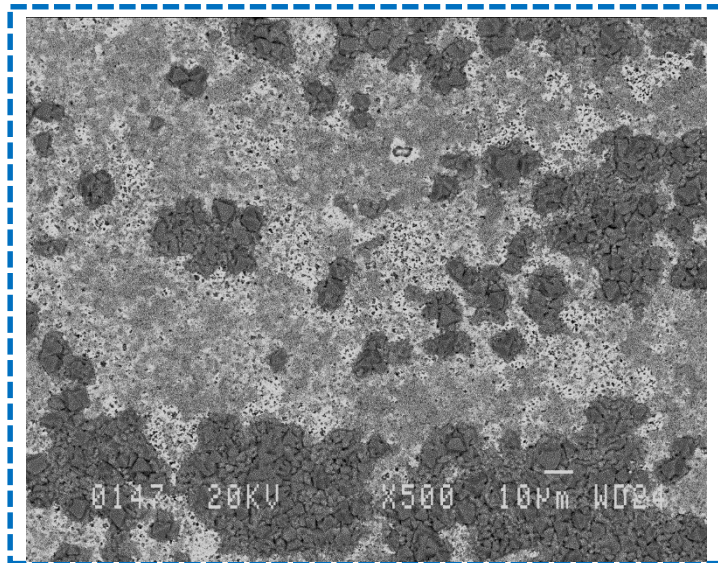
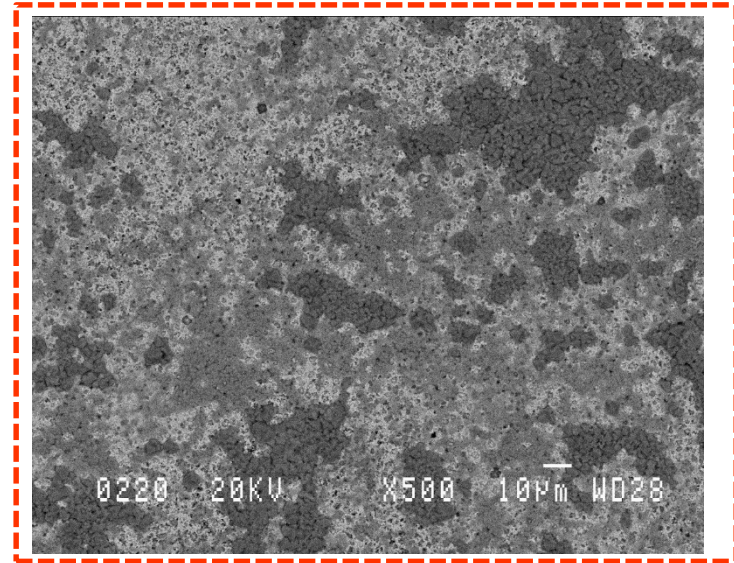
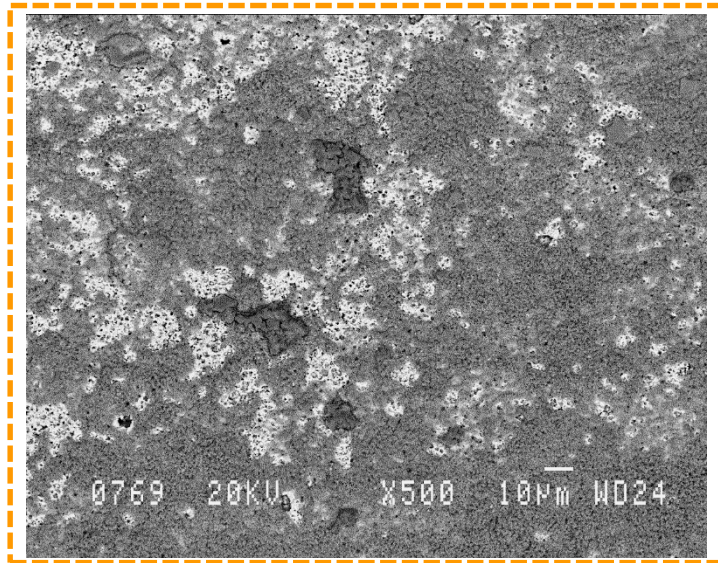
# Quantified Plots of Layer Thicknesses for All Transverse Sections



- No strong effect of increasing substrate roughness was observed.

# Comparison of Coated, Cycled, Plus Corroded Surfaces

Low – Est. Coating Thickness ( $\Delta D/2$ ) - High



Low - Substrate Roughness - High

- The oxide was attacked, but no open corrosion pits exposing the substrate were observed for the varied coated conditions.

# Summary of Results

- We prepared LSHR and ME3 specimens having varied surface finishes with average roughness values of 0.24  $\mu\text{m}$  to 1.50  $\mu\text{m}$ :  
**This correlated with coated, post-oxidation average roughness.**
- They were sputter coated with a NiCrY coating to varying estimated thicknesses ( $\Delta D/2$ ) from 7.6  $\mu\text{m}$  to 17.3  $\mu\text{m}$ :  
**Actual coating thicknesses in cross sections were more comparable than estimated by change in outer dimensions.**
- They were subjected to 500 or 1,020 thermal cycles from 25  $^{\circ}\text{C}$  to 760  $^{\circ}\text{C}$ ; then coating, outer oxide, and inner finger oxide depths were measured in cross sections:  
**No linear cracking or peeling was observed, with the protective coating layer still continuous and mostly intact under consistent oxide layers that did not strongly vary with roughness.**
- Specimen sections were corroded in air at 760  $^{\circ}\text{C}$  for 50 h:  
**The oxide was attacked, but no open corrosion pits exposing the substrate were observed for the varied coated conditions.**



# Conclusions

- **Substrate Roughness:**

A range of surface conditions and roughnesses on disk surfaces can be consistently sputter coated: low stress ground + polished (low roughness and cold work) up to strongly shot peened (high roughness and cold work).

- **Coating Thickness:**

Actual coating thicknesses for such different surface conditions can be maintained and consistent, however, estimation of coating thickness based on change in average outer dimensions can be tricky for varied surface roughness.

- **Thermal Cycling:**

The coating for such varied substrate conditions can still consistently resist cracking and enhanced cyclic oxidation during thermal cycling.

- **Corrosion Resistance:**

This thermally cycled coating can still provide protection of the superalloy from corrosion attack and pitting.

- **Next Steps:** More intervals of thermal cycles, mechanical fatigue, ?